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13. ABSTRACT (Maximum 200 words) A high-resolution x-ray diffractometer (HRXRD) with triple axis diffraction capabilities, combined in a single unit with a photoluminescence (PL) system has been acquired. This instrument combines complementary structural and optical characterization methods that will enhance ARO-funded research involving growth and defect-control studies of highly lattice-mismatched III-V/IV heterostructures. The HRXRD capabilities include double and triple axis diffraction, asymmetric and grazing incidence reflectivity, x-ray topography, and fluorescence. The PL capabilities include variable temperature measurements and lateral mapping of optical properties. PL and HRXRD analyses are possible at the same position, allowing for correlation between optical and structural properties. This system is installed and operational in a dedicated facility for this purpose. Measurements to understand how defect control methodologies impact properties of lattice-mismatched heterostructures are underway. For our ARO-supported research involving III-V growth and integration onto Si via relaxed compositionally-graded GeSi buffers, this instrument is being used to investigate the outstanding issues related to mismatched heteroepitaxy of III-arsenides, III-phosphides, LTG (low temperature grown) III-V's and on group IV substrates. These include quantifying the degree and distribution of lattice strain and relaxation in multiple layers, lattice tilt, composition, bandgap, interface and surface roughness, thermal expansion effects, mosaic spread, etc.			
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**ACQUISITION OF A TANDEM HIGH-RESOLUTION X-RAY
DIFFRACTOMETER-PHOTOLUMINESCENCE SYSTEM (DURIP)**

FINAL REPORT

for

Grant no. DAAD19-00-1-0035

(OSURF Project no: 739024)



10/8/91

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Executive Summary

A unique instrument capable of performing both high-resolution x-ray diffraction measurements and photoluminescence measurements on complex semiconductor heterostructures and nanostructures has been obtained with funds provided by this DURIP project. The system, assembled by Bede Scientific Inc, arrived in late 2000 and required some successful modifications in order to meet specifications. The manufacturer has been fully cooperative in this and we are quite pleased with the results. The instrument is housed in our Interdisciplinary Laboratory for Nanoscale Measurements in the Department of Electrical Engineering. Details of the equipment and a summary of research projects that will use this instrument are described below.

Equipment List

Purchased from Bede Scientific, Inc. 14 Inverness Drive East, Englewood, CO 80112

High-Resolution X-Ray Diffractometer -- Variable Temperature PL Mapping System:

1. D1 system diffractometer, comprised of:
Goniometer, beam conditioner axes and crystals, EDRA
Detector with slits for reflectometry, Minicam control
interface, instrument control and data acquisition software,
Spellman 3kW x-ray source, and integral safety enclosure \$135,000
2. Precision slit set, for high precision reflectivity and alignment \$2,000
3. Parabolic mirror for high intensities \$15,000
4. 100mm five axis scanning sample stage, with:
100mm xy scanning, 360° azimuthal rotation,
6mm z translation, and 90° tilt \$23,000
5. Combination Triple Axis and Parallel Beam Optics stage
with 0.4° graphite monochromator \$36,000
6. Radiation monitoring unit \$500
7. PC for control and data acquisition \$3,000
8. Bede Lab analytical software suite (RADS, REFS,
Mercury module, Contour, and Peaksplit) \$0
(donation: value \$39,500)
9. Bede photoluminescence system: includes wide-band fiber
optic coupling between laser/multiple grating (200 – 1800 nm) spectrometer
unit and specimen stage, Ar⁺ laser, PMT and InGaAs detectors \$120,000
10. Anton-Paar TTK-450 heating/cooling environmental stage,
-193 to +450°C, in vacuum or inert gas \$43,500

TOTAL CAPITAL EQUIPMENT COST

\$378,000

Research projects involving this acquisition

Research work described in proposal:

Two ARO programs were the primary, initial projects impacted by this instrument:

1. Program Title: Device-Quality Highly Mismatched Semiconductor Materials Grown on Si Substrates Using Unique Dislocation Engineering
PI: Steven A. Ringel
DoD Org.: Army Research Office – Materials Sciences Division (monitor - Dr. John Prater)
Grant no.: DAAG55-97-1-0111
2. Program Title: Investigation of Non-Stoichiometric III-V Materials and Heterostructures Grown on Ge/GeSi/Si Virtual Substrates
PI: Steven A. Ringel
DoD Org.: Army Research Office – Materials Sciences Division (monitor - Dr. John Prater)
Grant No.: DAAG55-98-1-0142

This instrument is extremely versatile in its ability to provide information for multiple research programs. It was acquired with the intent of enhancing work being performed on the above programs initially, both of which involve growth and defect control studies of highly lattice-mismatched III-V/IV heterostructures that can enable integration of optoelectronic devices with Si technology. The presence of lattice mismatch, compositionally graded buffer interlayers and complex strain relaxation mechanisms are the primary reasons for the application of this instrument for these programs. As we have now assembled and tested the unit, we will be performing extensive XRD and PL studies of III-As and III-P based heterostructures grown on graded GeSi substrates. Among the many research items that we will investigate, one that will take complete advantage of our new capability is the important issue of strain related to thermal expansion coefficient differences and how this can be controlled via introduction of the appropriate amount of mismatch strain. This will require the ability to map asymmetric strain, analyze reciprocal space (k-mapping) and do so over a wide range of precision x-ray wavelengths. This will be performed on samples grown in our MBE facility that will be comprised of III-V overlayers ranging from InGaP and

GaAs/AlGaAs to InAsP and low-temperature-grown (LTG) III-V's so that we can assess the full structural science of our approach to electronic materials integration. The photoluminescence capability will be invaluable as it will provide spatial mapping of bandgaps so that another independent variable related to structural properties can be ascertained that also happens to be extremely relevant to the eventual fabrication of optoelectronic devices using these materials. The equipment will be a central tool for this and many similar sub-projects that are part of our larger goal to achieve device quality integration of various III-V devices with Si.

Other Research of Interest to DoD

In addition to these two core ARO programs, we expect the XRD-PL system to be used for other DoD programs as well. These include several ONR programs (J. Zolper, monitor) that focus on defects in GaN and related nitride materials led by Ringel, and others led by L.J. Brillson in our department also from ONR (C. Wood, monitor) that also focus on defects in nitrides. For these programs, understanding the strain state and lateral inhomogeneities in structural properties as a function of growth conditions, substrate, layer composition, etc., is important to relate the presence of specific defects with structural properties and sources. Hence this equipment will provide important augmentation to these programs as well.

Finally, Ringel leads several other programs in the area of lattice mismatched growth, funded by both industry and other non-DoD agencies. The focus of those programs is on understanding grading in InAsP, InAlAs, and related material systems on InP, which have implications for devices working in the infrared spectrum. Issues similar to those of the core ARO programs are present here as well, and we expect the XRD-PL system to be used extensively to understand the structural properties of these heterostructures.

Summary

This DURIP award was used to procure a unique instrument that combines state of the art high-resolution XRD research with photoluminescence mapping. The instrument has been received and is functioning to specification. We are currently initiating research with the instrument for both the core ARO programs as well as other programs that involve lattice-mismatched heteroepitaxy of electronic material heterostructures.